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Butter — Determination of firmness

Beurre — Détermination de la fermeté

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16305 | IDF 187 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF), in collaboration with AOAC International. It is being published jointly by ISO and IDF and separately by AOAC International.

Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO and AOAC International in the development of standard methods of analysis and sampling for milk and milk products.

Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50 % of the National Committees casting a vote.

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All work was carried out by the Joint ISO/IDF/AOAC Action Team, *Physical properties and rheological tests*, of the Standing Committee on *Minor components and characterization of physical properties*, under the aegis of its project leader, Mr G.J. Beutick (NL).

Butter — Determination of firmness

1 Scope

This International Standard specifies a method for the determination of the firmness of butter.

The method is also applicable to butter prepared by recombination of milk components, aeration of butter and butter to which vegetable fat, spices or other foods have been added. Any changes with regard to the preparation of the butter, however, will influence its firmness characteristics. Therefore for the purposes of this International Standard, these products are not included.

NOTE The firmness of butter as determined by this method is correlated with the spreadability of butter as determined by a test panel (see Reference [4]).

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
firmness of butter
force needed to cut through a sample cube of butter using a wire in accordance with the procedure specified in this International Standard

NOTE The firmness of butter is expressed in newtons (N).

3 Principle

A butter sample, at a strictly specified temperature, is cut vertically through using a defined horizontal cutting wire that moves downwards at a constant speed. The cutting force is measured in newtons by a force-measuring instrument.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Incubator, capable of operating at 10 °C ± 1 °C.

4.2 Water bath, capable of operating at 10 °C ± 0,1 °C.

4.3 Sample holder, with dimensions as given in Figure A.1, of accurate measuring size of 25 mm ± 0,2 mm, fitted with a (replaceable) handle (see Figure A.2) or a U-shaped handle (see Figure A.3).

4.4 Probe, of metal, with a cutting wire stretched tight for the measurement (see Figure A.4).

The cutting wire shall be made of stainless steel, and shall be round with diameter of 0,3 mm ± 0,01 mm.

4.5 Measuring apparatus (e.g. texture analyser), capable moving the probe vertically downwards at a constant speed of 1,0 mm/s and parallel to one edge of the test cube.

It shall be capable of measuring the force over an appropriate measuring range. It shall be connected to a computer for continuous measuring of the force with an error of less than 0,05 N at 2 N and of at least 20 data points per second, placed in a cooling chamber or cooling device (4.7).

Note that heat produced by the measuring apparatus may effect the firmness measurements if no appropriate measures are taken.

4.6 Sample preparation tool (e.g. a clip), with limbs of length approximately 70 mm, comprising a stretched tight wire, made of stainless steel, of maximum diameter 0,5 mm and length of about 100 mm, or a stopping knife with a blade not thicker than 0,5 mm.

4.7 Cooling device, for example that shown in Figure A.5.

5 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707.

Test samples taken directly after production usually have a temperature above 10 °C and shall be cooled to or below that temperature. The temperature during transportation shall be kept at or below 10 °C.

6 Storage of test samples

Store test samples taken directly after production for (official) control purposes in an incubator (4.1) at 10 °C ± 0,1 °C for (10 ± 1) days. If it is necessary to delay or interrupt this storage time, cool the test samples to a temperature of between 0 °C and 4 °C for up to 4 days, or by freeze them to a temperature of below −20 °C for a longer period.

NOTE 1 The setting of butter is still evolving after its production and is strongly related to the storage temperature: the higher the temperature, the faster the setting. When stored at 10 °C, it can take up to 30 days before the setting stabilizes, while after 10 days about 80 % of the final firmness has already been reached. The butter setting is slow at a temperature of between 0 °C and 4 °C. Under these conditions no difference in the firmness of butter can be measured for up to 4 days after production. The setting stops completely when the butter samples are frozen.

The time of storage at 10 °C ± 1 °C for test samples with another or unknown storage history (storage temperature/time combination) shall be agreed upon by the parties concerned. The conditions agreed upon shall be noted in the test report.

NOTE 2 For example, this might be the case when the firmness of a test sample derived from consumer packages is to be measured.

7 Checking the cutting wire

Check the length of the cutting wire regularly as it might stretch out as a result of intensive use, which also might affect the precisely specified diameter of the cutting wire. Check the wire length by using one of the following techniques.

a) Hang a weight of 200 g in the middle of the cutting wire. Measure the deviation of the wire from its original position by using an appropriate measuring instrument.

b) Set the measuring apparatus (4.5) to a cut-off force of 2 N. Let the apparatus measure the deviation in the central position of the metal clip as it is pushed against a round metal rod with diameter of approximately 0,5 cm.

In both cases, the deviation of the central position shall not exceed 1 mm for a cutting wire with a length of 5 cm.

8 Procedure

8.1 Preparation of test portion

Immediately before putting the test portions in the water bath, cut, without any delay, two test portions from the (incubated) test sample (Clause 6) by means of the sample holder (4.3).

Carefully press the used sample holder (4.3) into the test sample until it is completely filled up with the test portion. Cut off that part of the test portion that runs out of the sample holder with the sample preparation tool (4.6) in accordance with the instructions for the used sample holder (see Annex A).

Prevent any deformation of the test portion in the sample holder during its preparation.

If a room is not available with a temperature of 10 °C ± 1 °C, prepare the two test portions as quickly as possible to avoid warming.

8.2 Temperature equilibration of the test portions

Equilibrate the temperature of the two test portions to the measuring temperature of 10 °C ± 0,1 °C by keeping the sample holder containing the test portions in the water bath (4.2) for at least 1 h, with a maximum of 5 h, before starting the measurement.

8.3 Temperature control during the measurements

The temperature of the test portions during the measurements of the firmness shall not exceed 10 °C ± 0,2 °C. Preferably, determine the firmness in a cooling chamber or using a cooling device (4.7).

8.4 Determination

Place the test portion in the measuring apparatus (4.5). Fix the cutting wire of the probe (4.4) exactly above the slot in the sample holder. Start the measurement.

The measuring apparatus (4.5) continuously records the measured force, as a function of time, until a cutting distance of at least 18 mm is obtained.

9 Calculation and expression of results

9.1 Calculation

The firmness of a test portion, expressed in newtons (N), corresponds to the arithmetical mean of the force values measured between 8 mm and 16 mm in cutting distance.

Check the data for internal consistency in the relevant region of between 8 mm to 16 mm. Results are acceptable when the difference between the minimum and the maximum force is less than 10 % of the calculated arithmetical mean of the firmness.

9.2 Expression of results

Express the result of the firmness of a test sample, expressed in newtons (N), as the mean of the results of the two test portions.

Round the results to two decimal places.

10 Precision

10.1 Interlaboratory test

Details of the interlaboratory test on the precision of the method are summarized in Annex B. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those specified in the Scope.

10.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 10 % (relative) of the arithmetic mean of the two results.

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 25 % (relative) of the arithmetic mean of the two results.

11 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the incubation period at 10 °C ± 1°C, or the conditions agreed between the parties;

d) the test method used, with reference to this International Standard;

e) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

f) the test result(s) obtained and, if the repeatability has been checked, the final quoted result obtained.

Annex A
(normative)

Apparatus

A.1 General sample holder

The sample holder shall be as described in 4.3 and shown in Figure A.1.

Dimensions in millimetres

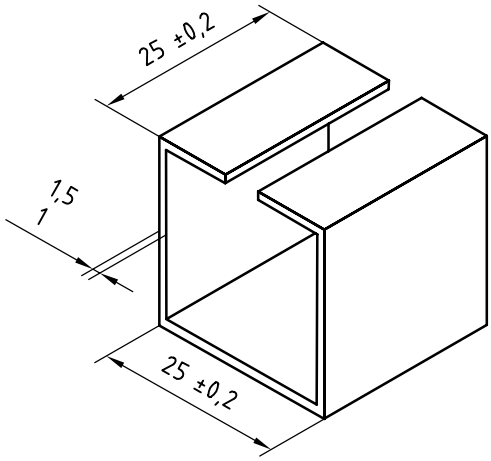


Figure A.1 — Sample holder

A.2 Sample holder fitted with a handle

Take care to prevent canting of this sample holder (see Figure A.2) during cutting and to prevent a cut that is not carried out vertically.

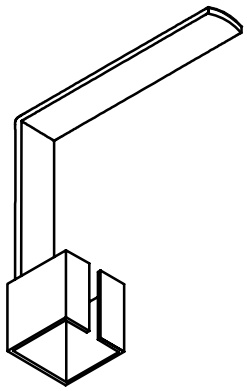


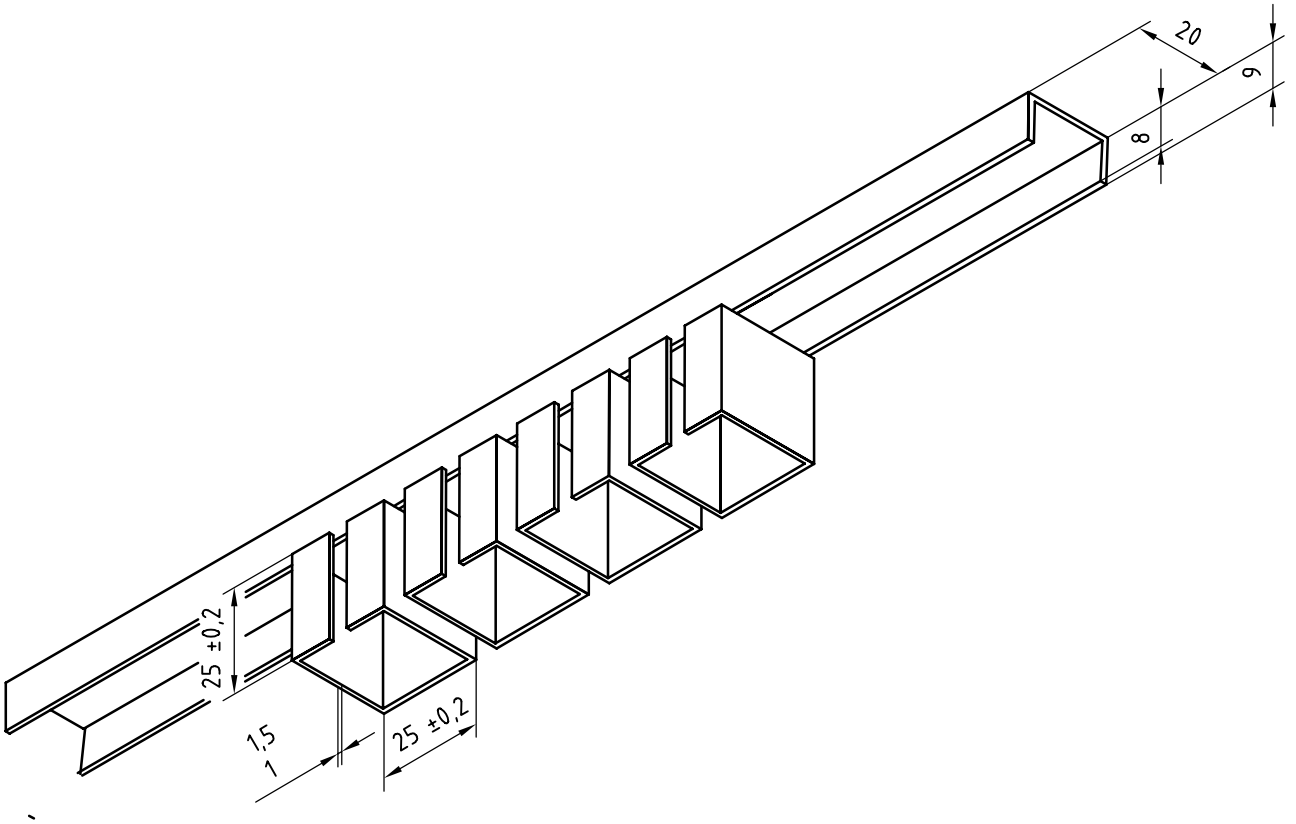
Figure A.2 — Sample holder fitted with a handle

A.3 Sample holder with a U-shaped handle

The use of a U-shaped handle to press the holder into the sample (see Figure A.3) has also proved to be successful for cutting test portions. Use of this device gives an even cutting of test portions and it may also be used for several cuttings at the same time.

Put the sample holder(s) on the test sample and push the holder(s) into the sample by means of the U-shaped handle until the holder(s) is (are) completely filled with sample. Remove the sample holder(s) using the sample preparation tool (4.6), without deformation of the inside of the test portion(s).

Dimensions in millimetres



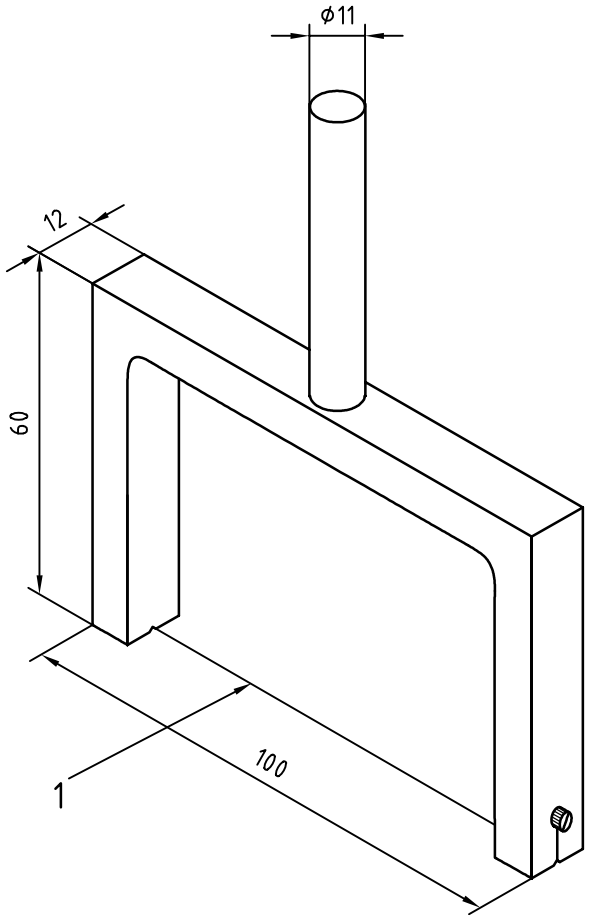
NOTE All dimensions are ± 0,2 mm.

Figure A.3 — Sample holder with a U-shaped handle

A.4 Probe

See Figure A.4.

Dimensions in millimetres



Key

1 cutting wire (∅ 0,3 mm ± 0,01 mm)

Figure A.4 — Probe

A.5 Cooling device

See Figure A.5.

Dimensions in millimetres

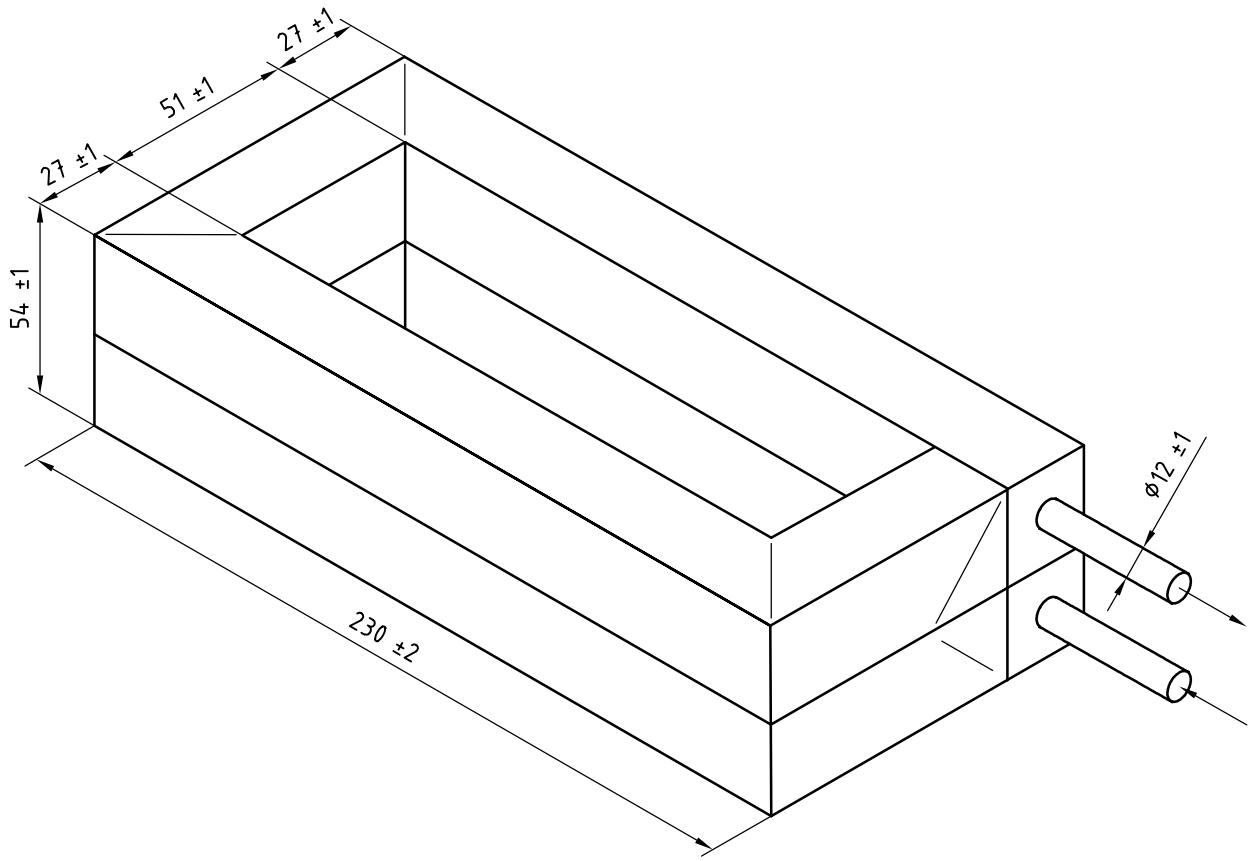


Figure A.5 — Example of a cooling device

Annex B
(informative)

Results of interlaboratory trial

An international collaborative trial involving 10 laboratories from 6 countries was carried out on 6 samples with different firmness. The 6 test samples were divided into 12 blind duplicated samples. The test was organized by COKZ (NL). The values are expressed in newtons (N) or as percent (relative).

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in Table A.1.

Table B.1 — Results of interlaboratory trial

	Sample					
	A	**B**	**C**	**D**	**E**	**F**
Participants after eliminating outliers	8	8	8	8	8	8
Mean value (N)	2,071	1,971	1,347	1,230	0,710	1,298
Repeatability standard deviation, s_r (N)	0,067	0,078	0,056	0,028	0,032	0,051
Coefficient of variation of repeatability limit (%)	9,1	11,1	11,7	6,5	12,6	11,1
Repeatability limit, r (= 2,8 s_r) (N)	0,188	0,219	0,158	0,080	0,090	0,144
Reproducibility standard deviation, s_R (N)	0,162	0,084	0,146	0,082	0,079	0,136
Coefficient of variation of reproducibility limit (%)	21,9	12,0	30,3	18,7	30,9	30,0
Reproducibility limit, R (= 2,8 s_R) (N)	0,454	0,236	0,408	0,230	0,221	0,389

Bibliography

[1] ISO 707 [1)], *Milk and milk products — Guidance on sampling*

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4] ROHM, J. *Journal of Texture Studies*, **20**, 1989

1) Equivalent to IDF 50.

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